

07/25/00
JC893 U.S. PRO

07-26-00

A

Please type a plus sign (+) inside this box → ☐

PTO/SB/05 (1/98)
Approved for use through 09/30/2000. OMB 0651-0032
Patent and Trademark Office: U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995 no persons are required to respond to a collection of information unless it displays a valid OMB control number.

UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 CFR 1.53(b))

Attorney Docket No. JJ-124B-R&D
First Inventor or Application Identifier Michael Francis Dube
Title Method of Improving Flavor in Smoking Article
Express Mail Label No. EI 519 567 967

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

ADDRESS TO: Assistant Commissioner for Patents
Box Patent Application
Washington, DC 20231

1. ☒ * Fee Transmittal Form (e.g., PTO/SB/17)
(Submit an original, and a duplicate for fee processing)
2. ☒ Specification [Total Pages 15]
(preferred arrangement set forth below)
 - Descriptive title of the invention
 - Cross References to Related Applications
 - Statement Regarding Fed sponsored R & D
 - Reference to Microfiche Appendix
 - Background of the Invention
 - Brief Summary of the Invention
 - Brief Description of the Drawings (if filed)
 - Detailed Description
 - Claim(s)
 - Abstract of the Disclosure
3. ☐ Drawing(s) (35 U.S.C. 113) [Total Sheets 0]
4. Oath or Declaration [Total Pages 4]
 - a. ☒ Newly executed (original or copy)
 - b. ☐ Copy from a prior application (37 C.F.R. § 1.63(d))
(for continuation/divisional with Box 17 completed)
[Note Box 5 below]
 - i. ☐ DELETION OF INVENTOR(S)
Signed statement attached deleting inventor(s) named in the prior application, see 37 C.F.R. §§ 1.63(d)(2) and 1.33(b).
5. ☐ Incorporation By Reference (useable if Box 4b is checked)
The entire disclosure of the prior application, from which a copy of the oath or declaration is supplied under Box 4b, is considered to be part of the disclosure of the accompanying application and is hereby incorporated by reference therein.

6. ☐ Microfiche Computer Program (Appendix)
7. Nucleotide and/or Amino Acid Sequence Submission (if applicable, all necessary)
 - a. ☐ Computer Readable Copy
 - b. ☐ Paper Copy (identical to computer copy)
 - c. ☐ Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

8. ☒ Assignment Papers (cover sheet & document(s))
 9. ☐ 37 C.F.R. §3.73(b) Statement (when there is an assignee) ☐ Power of Attorney
 10. ☐ English Translation Document (if applicable)
 11. ☐ Information Disclosure Statement (IDS)/PTO-1449 ☐ Copies of IDS Citations
 12. ☐ Preliminary Amendment
 13. ☒ Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
 - * Small Entity Statement(s) ☐ Statement filed in prior application. Status still proper and desired (PTO/SB/09-12)
 14. ☐ Certified Copy of Priority Document(s) (if foreign priority is claimed)
 15. ☐ Other:
 16. ☐ Other:
- * A new statement is required to be entitled to pay small entity fees, except where one has been filed in a prior application and is being relied upon.

17. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in a preliminary amendment:

☐ Continuation ☐ Divisional ☐ Continuation-in-part (CIP) of prior application No: _____

Prior application information: Examiner _____

Group / Art Unit: _____

18. CORRESPONDENCE ADDRESS

☐ Customer Number or Bar Code Label

(Insert Customer No. or Attach bar code label here)

or ☒ Correspondence address below

Name	Grover M. Myers				
	Law Department - Patents				
Address	R.J. Reynolds Tobacco Company				
	Bowman Gray Technical Center, P.O. Box 1487, 950 Reynolds Boulevard				
City	Winston-Salem	State	NC	Zip Code	27102
Country	USA	Telephone	(336) 741-2694	Fax	(336) 741-5449

Name (Print/Type)	Grover M. Myers	Registration No. (Attorney/Agent)	25,359
Signature	<i>[Signature]</i>	Date	7/25/00

Burden Hour Statement: This form is estimated to take 0.2 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Box Patent Application, Washington, DC 20231.

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

Certificate of Mailing under 37 CFR 1.8

I hereby certify that this correspondence is being deposited with the United States Postal Service with sufficient postage as first class mail in an envelope addressed to:

Assistant Commissioner for Patents
Washington, D.C. 20231

on July 25, 2000
Date

Parthenia S. Galloway
Signature

Parthenia S. Galloway
Typed or printed name of person signing Certificate

Note: Each paper must have its own certificate of mailing, or this certificate must identify each submitted paper.

- | | |
|---|---------|
| 1. Utility Patent Application Transmittal | 1 page |
| 2. Fee Transmittal | 2 pages |
| 3. Declaration for Utility or Design | 4 pages |
| 4. Recordation Form | 4 pages |
| 5. Assignment | 3 pages |

APPLICATION FOR UNITED STATES LETTERS PATENT

TITLE: METHOD OF IMPROVING FLAVOR
IN SMOKING ARTICLE

INVENTOR: MICHAEL FRANCIS DUBE
LUIS MAYAN DOMINGUEZ
CHI-KUEN SHU
ELWOOD LEE WILLIAMS, JR.
KENNETH WAYNE SMITH
WILLIAM MONROE COLEMAN, III

ASSIGNEE: R. J. Reynolds Tobacco Company
Winston-Salem, North Carolina

Number EI 519 567 967

Date of Deposit July 25, 2000

I hereby certify that this paper or fee is
being deposited with the United States Postal
Service "Express Mail Post Office to Addressee"
service under 37 CFR 1.10 on the date indicated
above and is addressed to the Commissioner of
Patents and Trademarks, Washington, D.C. 20231.

Parthenia S. Galloway

(Typed or printed name of person mailing
paper or fee)

Parthenia S. Galloway

(Signature of Person mailing paper or fee)

Grover M. Myers, Esquire
Reg. No. 25,359
R. J. Reynolds Tobacco Company
P.O. Box 1487
Winston-Salem, NC 27102-1487
Tel: 336/741-2694

METHOD OF IMPROVING FLAVOR IN SMOKING ARTICLE

Field of the Invention

The present invention generally relates to smoking articles such as cigarettes, and in particular to smoking articles with improved flavor and aroma and process for making same.

Background Of the Invention

Popular smoking articles, such as cigarettes, have a substantially rod shaped structure and include a charge of smokable material such as strands or shreds of tobacco (e.g., tobacco cut filler) surrounded by a paper wrapper thereby providing a so-called "tobacco rod."

During smoking, the tobacco cut filler in the tobacco rod is heated and burned producing smoke. The flavor and aroma of the smoke are important characteristics of smoking articles. In conventional cigarette manufacturing processes, a large amount of sugar is typically applied as a casing ingredient to tobacco leaf blends, especially air-cured blends, before cutting. Conventionally, this casing process involves applying sugar as a solution and then heating the sugar-treated tobacco at a relatively high temperature. Normally a portion of the sugar is caramelized, i.e., undergoes Browning reactions, during the heating step, thus generating caramel-like compounds. Some of these caramel compounds exhibit flavors which are believed to be important in masking the bitterness in the smoke and improving the flavor of the smoking article.

The term "caramel" is generally used to designate a group of a large number of complex molecules with different chemical properties. Several types of caramel are known in the art including plain caramel, caramel produced by ammonia process or caustic process, and caramel produced by an ammonium sulphite process. The color and

flavor can vary somewhat with the different types of caramel. Caramel normally exhibits a brown color and the darkness of the brown color of a particular caramel preparation can vary depending on the caramel preparation process. Normally, caramel produced by either the ammonia process, the caustic process, or the ammonium sulphite process, exhibits darker colors and is especially suited as coloring agent.

Various caramel compositions have been used or proposed for use as components of cigarettes. For example, caramel has been used as a coloring agent for cigarette wrappers. *See* U.S. Patent No. 5,284,166. Caramel has also been used in the substitute fillers of substitute cigarettes as a coloring agent and to simulate natural tobacco material. *See* U.S. Patent Nos. 3,934,594 and 3,929,141, and France Patent No. 2,767,649.

Although it is conventionally understood that sugar is incorporated into tobacco as a casing component, it is caramelized during the manufacturing process and during smoking. In many cases, the complex chemistry of tobacco can interfere with the caramelization process. In particular, the sugar can react with other components of the tobacco materials in the smoking article including carboxylic acids and nitrogen sources. As a result, the yield of the desirable sweet and caramel-like flavor materials generated *in situ* by sugar caramelization is usually low. Moreover, since the conventional casing heating step does not caramelize or otherwise convert all of the applied sugar into flavorant compounds, the remaining sugar is heated during smoking in the smoking article, but the heating process applied to tobacco during smoking is not optimum for increasing the yield of desirable caramel-like flavorants.

SUMMARY OF THE INVENTION

The present invention provides for a method of improving the flavor and aroma characters of a smoking article by heating a sugar optionally in admixture with a hydroxide to generate a flavorful caramel which is essentially free of any nitrogen-containing caramel compounds, and the caramel composition is applied to tobacco materials that are used to provide a smoking article.

In particular, in accordance with the present invention, a mixture is provided containing a reducing sugar and optionally a hydroxide. Typically, the mixture is in an aqueous form. A hydroxide of an alkali metal including sodium hydroxide, potassium

hydroxide, and the like is preferably included in the mixture. Normally, the mixture does not contain any significant amount of amino or other components that are capable of reacting with the reducing sugar so as to substantially interfere or compete with the caramelization of the reducing sugar or to generate a substantial amount of non-caramel flavor substances.

The mixture is exposed to a temperature sufficiently high and for a period of time sufficiently to provide a flavorful caramel composition. Normally, heat treatment is conducted at a temperature of at least about 90°C, preferably at least about 100°C, more preferably at least about 120°C. The resultant flavorful aqueous composition contains a substantial amount of flavorful caramel compounds including furaneol, maltol, cyclotene, α -Angelica lactone, and the like. In addition, the flavorful caramel composition also contains unreacted reducing sugar.

The flavorful caramel composition can be employed in improving the flavor characteristics of smoking articles such as cigarettes. In particular, the flavorful composition is applied directly to tobacco materials as a casing component or top dressing ingredient. The flavorful composition is especially suited for application to tobacco leaves or tobacco cut fillers. Smoking articles made from the tobacco leaves or tobacco cut fillers exhibit significantly improved flavor that are pleasant, sweet, and caramel-like.

In the method of the present invention, the caramelization of sugars is conducted externally from the tobacco, and in a controlled environment that can be optimized for the caramelization reaction, preferably in an enclosed environment under pressure, and in the absence of interference from other chemical compounds such as nitrogen sources. As a result, a greater amount of caramel-like flavorants can be generated from a relatively small amount of sugar. In addition, the flavorful caramel composition employed in this invention is essentially free of nitrogen-containing substances and therefore exhibits desirably pure pleasant, sweet, and caramel-like flavor. In addition, the caramel composition contains a lower content of undesirable caramel species and solid carbon precipitates and black pigments, and thus exhibits improved color properties. The flavorful caramel composition can be applied to tobacco materials as a casing or top dressing ingredient, so that other materials typically used in conventional casing can be

reduced or even omitted from the smoking article of the present invention. The resultant smoking article exhibits unique pleasant flavor.

The foregoing and other advantages and features of the invention, and the manner in which the same are accomplished, will become more readily apparent upon consideration of the following detailed description of the preferred embodiments of the invention.

DETAILED DESCRIPTION OF THE INVENTION

This invention provides for a method of improving the flavor characters of a smoking article by heating an essentially nitrogen-free mixture containing a reducing sugar and optionally a hydroxide-containing base compound to generate a flavorful composition and applying the composition to tobacco materials in the smoking article.

Suitable reducing sugars that can be used in the invention include, but are not limited to, glucose, fructose, sucrose, mannose, galactose, rhamnose, and mixtures thereof. The reducing sugar can be in a pure form or in unrefined form, e.g., high fructose corn syrup (HFCS) which has a high content of fructose. Other derivatives of reducing sugars can also be used, e.g., phosphate-substituted reducing sugars (e.g., glucose-6-phosphate, fructose-6-phosphate, and fructose-1,6-diphosphate). Reducing sugars can also be provided in the form of precursors that can readily release reducing sugars under the reaction conditions employed in the method of this invention. Examples of suitable precursors can include disaccharides and polysaccharides, and derivatives thereof. In such cases, reducing sugars can be generated by the hydrolysis of disaccharides or polysaccharides. In a preferred embodiment of the invention, a high fructose corn syrup (HFCS) having at least about 30% by weight of fructose is used as the reducing sugar component. For example, a high fructose corn syrup having about 42% by weight of fructose is commercially available from Corn Products International, Bedford Park, IL.

Optionally, the mixture also contains a strong non-amino-base compound, preferably an alkali metal hydroxide such as sodium hydroxide, potassium hydroxide, and the like. It has been discovered that alkali hydroxide can increase the yield of flavorful caramel components without causing the formation of a substantial amount of

undesirable insoluble solid materials in the resultant flavorful aqueous composition as can be generated by, e.g., ammonium hydroxide or ammonium carbonate. Preferably, sodium hydroxide is used.

The mixture typically does not contain any significant amount of chemicals that will substantially interfere with the caramelization reactions of the reducing sugars. Preferably, the mixture does not contain any significant amount of amino compounds or other components that are capable of reacting with the reducing sugars to generate nitrogen-containing caramels or compounds other than caramel components. For example, it has been observed that when ammonium compounds such as diammonium phosphate is included in the mixture, not only non-caramel flavor substances are generated after heat treatment, but also the resultant composition contains an undesirable amount of solid carbon precipitates or black pigments.

Three general kinds of caramel are known in the art: (1) plain caramel, (2) caramel produced by ammonia process or caustic process, (3) caramel produced by an ammonium sulphite process. The heat treatment of the mixture of this invention is intended to produce plain caramel but not the other two types of caramels. Accordingly, the mixture of the present invention preferably does not include ingredients such as ammonium compounds (e.g., ammonia, ammonium hydroxide, and ammonium salts such as diammonium phosphate), amino acids, and the like. Moreover, even though the caramelization process employed in the invention can involve use of an alkali metal hydroxide, the resultant caramel is substantially different from caustic process caramels because the level of the hydroxide in the mixture of the present invention is substantially lower than that used for making caustic caramels. The content of alkali metal hydroxide (calculated on a water-free basis) can be from about 0% to about 30%, preferably from about 0.5% to about 10% by weight based on the total weight of the mixture.

Generally, the total content of any undesirable reactive materials should be less than about 1% by weight based on the reducing sugar content, preferably less than 0.3% by weight, more preferably less than 0.10% by weight. Other known flavorants such as cocoa and licorice can be included in the mixture. However, in a preferred embodiment, the mixture contains only reducing sugar(s) and optionally sodium hydroxide.

005220" 02842960

In the reaction mixture, the content of reducing sugar can be from about 5% to about 80%, preferably from about 20% to about 60%, and more preferably from about 30% to about 50% by weight (dry weight basis) based on the total weight of the mixture.

The reaction mixture can be in a solid or liquid form. Typically the reaction mixture is in an aqueous form. The reducing sugar and optionally alkali metal hydroxide are mixed together with a liquid medium having an aqueous character such as a liquid consisting primarily of water, normally greater than about 90 weight percent water, and can be essentially pure water. For example, the liquid medium can be distilled water, tap water, or the like.

The caramelization reaction is conducted by subjecting the aqueous mixture to heat treatment for a time and under conditions sufficient to generate a flavorful caramel composition. The aqueous mixture can be heated to a temperature of at least about 90°C, preferably at least about 100°C, more preferably to a temperature of at least about 125°C. Advantageously, the heat treatment is conducted at a temperature of at least about 150°C or greater. However, it is desirable to subject the mixture to a temperature below temperatures causing an undesirable formation of components that are deleterious to the flavor characteristics of the flavorful caramel composition produced. Normally, the heat treatment is conducted at a temperature preferably below about 200°C.

The heat treatment of the mixture can be conducted under an ambient pressure environment, i.e., in a vented reactor. However, it is preferred that the mixture is heated in an enclosed pressure-controlled environment. Such an environment is provided by enclosing the mixture in an air sealed vessel or chamber. Typically, a pressure-controlled environment is provided using a pressure vessel or chamber which is capable of withstanding relatively high pressures. Preferred pressure vessels are equipped with an external heating source. Examples of vessels which provide a pressure controlled environment include reactors sold by American Reactor Corporation having a sealable vent and a heating jacket, a high pressure autoclave from Berghof America Inc., and a Parr Reactor Model No. 4522 from The Parr Instrument Co. Operation of such exemplary vessels will be apparent to the skilled artisan. Typical pressures experienced by the mixture during the process of the present invention range from about 10 psig to about 1,000 psig, normally from about 20 psig to about 500 psig. Pressures experienced

005220 0284360

by the mixture typically exceed 100 psig during the process of the present invention. The heat treatment of the mixture under pressure can be performed under an inert atmosphere. For example, nitrogen or argon gas can be employed in order to provide an inert atmosphere. However, the heat treatment can also be conducted under ambient air.

The amount of time required for the heat treatment can vary with the temperature. Typically, less time is necessary if the aqueous mixture is under a moderate heat treatment. Conversely, more time is required when the reacting temperature is low. Preferably the time and temperature are sufficient to cause reaction of at least about 5%, preferably at least about 10%, more preferably at least about 25% by weight of the starting sugars or precursors to be caramelized. Advantageously, at least about 50% of the sugars is caramelized. For example, satisfactory results are typically achieved when an aqueous mixture is heated at about 100°C for about 3 hours, or at about 175°C for about 10 minutes. Normally, it is desirable to control the time/temperature profile of the heat treatment so that the mixture is not subjected to a particularly high temperature for a lengthy period of time and the generation of materials deleterious to the flavor characteristics of the resultant flavorful composition is avoided. Some minor experiments may be required to determine the optional reacting time at a particular temperature, this being well within the capability of one skilled in the art once apprised of the present disclosure.

In addition, it is highly desirable to employ a pressure vessel equipped with an agitation mechanism such that the mixture experiences a relatively uniform temperature throughout the treatment period. In particular, it is highly desirable for the mixture to be heated uniformly throughout as much as possible at the maximum temperature to which the mixture is subjected.

The heat treatment of the mixture in the present invention typically leads to the caramelization of the reducing sugar, and the resultant flavorful composition contains a substantial amount of caramel components including, but not limited to, furaneol, maltol, cyclotene, α -Angelica lactone, and the like. The composition also contains a large amount of unreacted reducing sugar, typically at an amount of at least about 25% by weight based on the total weight of the composition. Typically the heat treatment results in a flavorful composition that is aqueous which does not contain a substantially amount

of solid aggregates. The aqueous composition may optionally be filtered, e.g., through a 60-mesh screen filter to remove large solid aggregates.

The flavorful composition thus prepared can be incorporated into smoking articles thus producing smoking articles containing "externally produced" caramel components. The term "externally produced" is used herein to refer to the caramel components produced externally independent of tobacco materials, i.e., caramel components that are neither produced on or in the tobacco during processing thereof nor generated *in situ* during smoking of the smoking article.

The flavorful composition produced in the method of this invention can be incorporated into smoking articles by various methods. The composition can be concentrated to reduce the water content and increase the content of useful flavorful compounds. Alternatively, the flavorful composition produced after heat treatment is used without additional processing.

In accordance with the present invention, the flavorful composition generated in the present invention is applied directly onto tobacco leaves or tobacco cut fillers made therefore as a casing material or top dressing ingredient. As is well known in the art, casing materials are used as additives to enhance the flavors in smokable materials. In cigarette manufacturing processes, casing materials are typically added to tobacco leaf blends before cutting. Casing materials are usually applied as suspensions or solutions. Exemplary casing ingredients that are commonly used in the art include, e.g., sugar, humectants such as glycerine or a higher glycol, licorice, cocoa, etc. The flavorful composition prepared in the present invention can be mixed with any of the conventional ingredients suspended or dissolved in the casing. The flavorful composition can also be used as the only casing ingredient. The casing can be applied to the leaf blend by either spraying or as a dip casing, or by other processes known in the art to allow the flavorful compounds in the composition to be coated onto or absorbed by the tobacco leaf blend.

The flavorful composition produced in the present invention is rich in flavorful caramel components as well as sugars. Accordingly, in advantageous embodiments of the invention, sugars and other ingredients added via conventional casing or like treatments can be reduced or eliminated. Advantageously, no additional casing ingredients are used other than the flavorful composition of the present invention.

The flavorful composition produced in accordance with the method of the present invention can also be incorporated into smoking articles as a top dressing ingredient. As is well known in the art, top dressing is added after the tobacco blend is cut into shreds or "cut fillers," to supply aroma or pleasing flavor. Top dressing is usually applied as a spray solution containing highly aromatic, perfume-like substances and a material such as a glycol to retard the evaporation of the flavorant in the cigarette or cigarette package. The flavorful composition can thus be used as a top dressing ingredient in the top dressing spray solution. The aqueous flavorful composition alone or in admixture with other conventional top dressing ingredients (e.g., humectants such as glycerol and the like, aroma-enhancing flavorants, etc.) can be sprayed onto the tobacco shreds or cut fillers. The composition can also be incorporated into smoking articles concurrently as the tobacco cut filler is formed into cigarette rods.

The tobacco leaf blends or cut fillers having the flavorful composition of this invention applied therein can also be used in a similar manner in many types of smoking articles other than the currently widely available cigarette constructions. For example, tobacco cut filler having the flavorful composition applied therein may be combined with aerosol forming materials, and employed in the manufacture of those smoking articles described in U.S. Patent Nos. 4,708,151 to Shelar; 4,771,795 to White et al.; 4,714,082 to Banerjee et al.; 4,756,318 to Clearman et al.; and 4,793,365 to Sensabaugh et al., as well as European Patent Publication Nos. 212,234 and 277,519, the disclosures of which are incorporated herein by reference. In addition, the tobacco cut filler can also be incorporated into those smoking articles described in U.S. Patent No. 5,074,321 and European Patent Publication No. 280,990.

The amount of the flavorful composition employed in smoking articles can vary. Generally up to 10-20% of the resulting liquid flavorful composition based on the dry weight of tobacco materials, can be applied to tobacco, including blends and tobacco components that are used to prepare cigarettes. Typically, the flavorful composition is applied at an amount of from about 5% to about 8% by weight based on the total dry weight of the tobacco leaf or cut filler in the smoking article. When a large amount of the flavorful composition needs be added, the tobacco leaves or cut filler can be dried to some extent to reduce the moisture content thereof before or after the liquid composition

is applied thereto. In this way, heating the impregnated tobacco leaves or cut filler to reduce moisture before packaging into cigarette rods can be minimized. Preferably, smoking articles, e.g. cigarettes prepared by the method of this invention contain at least the following caramel components: at least about 5 ppm of cyclotene, preferably at least about 10 ppm of cyclotene, at least about 7.5 ppm of furaneol and maltol, preferably at least about 25 ppm of furaneol and maltol wherein the caramel components are provided from the flavorful caramel composition prepared in this invention. When smoked, smoking articles prepared in accordance with the present invention exhibit pleasant, sweet, burn, and caramel-like flavors.

The following example is provided in order to further illustrate preferred aspects of the invention but should not be construed as limiting the scope thereof. Unless otherwise noted, all parts and percentages are by weight.

EXAMPLE I

100 grams of high fructose corn syrup (HFCS) (Corn Products International, Bedford Park, IL) containing 42% by weight of fructose was mixed thoroughly with 5 ml of NaOH solution (33% by weight) in a round bottom flask. The mixture was heated to and held at 100°C for three hours. The reaction mixture obtained was immediately cooled to room temperature using cold water. The resultant composition was tested for the contents of caramel components by the method described below. The result is shown in Table 1.

The furaneol, maltol, cyclotene, and α -angelica lactone contents were determined by a reversed-phase HPLC method with UV detection at 280 nm. The system used for these measurements was a Waters 2690XE Alliance HPLC system with a Waters 486 Tunable Absorbance Detector set at 280 nm. The autosampler was kept at 4°C at all times. The column employed for this separation was a Beckman ODS™, 5 μ m, C₁₈ reversed-phase column (4.6 x 250 mm). The mobile phase consisted of a gradient consisting of 90% deionized water with 0.005M KH₂PO₄ and 0.09M Acetic Acid, 10% methanol to 80% deionized water with 0.005M KH₂PO₄ and 0.09M Acetic Acid, 20% methanol in 10 minutes using a linear gradient. The gradient rate was thus 1%/min.

Samples were prepared by accurately weighing about 0.55 grams of a caramel cooked casing and dissolving in 5.0 ml of deionized water. This mixture was filtered through a Whatman PVDF Autovial-5™ directly into a Waters autosampler vial. Injection volume was 50 µl. The method could not resolve furaneol and maltol but measured their sum. A standard addition experiment demonstrated that the method was accurate as well as repeatable.

EXAMPLE 2

200 grams of high fructose corn syrup (HFCS) (Corn Products International, Bedford Park, IL) containing 42% by weight of fructose was mixed thoroughly with 10 ml of NaOH solution (33% by weight) in round bottom flask. The mixture was heated to and held at 175°C for 10 minutes. The reaction mixture obtained was immediately cooled to room temperature using cold water. The resultant composition was tested for the contents of caramel components by the method described in Example 1. The result is shown in Table 1.

EXAMPLE 3

200 grams of high fructose corn syrup (HFCS) (Corn Products International, Bedford Park, IL) containing 42% by weight of fructose was mixed thoroughly with 10 ml of NaOH solution (33% by weight) in round bottom flask. The mixture was heated to and held at 125°C for 10 minutes. The reaction mixture obtained was immediately cooled to room temperature using cold water. The resultant composition was tested for the contents of caramel components by the method described in Example 1. The result is shown in Table 1.

Table 1

Sample	Furaneol + Maltol (ppm)	Cyclotene (ppm)	α -Angelica Lactone (ppm)
Example 1	25	0.0	3.0
Example 2	327.0	108.0	17.0
Example 3	86.0	45.0	7.0
DDW 424*	75.0	21.0	4.0
DDW 525C-1*	44.0	16.0	31.0

* Caramel products purchased from D.D. Williamson & Co., Inc., Louisville, KY

Many modifications and other embodiments of the invention will come to mind to one skilled in the art to which this invention pertains having the benefit of the teachings presented in the foregoing descriptions and the associated drawings. Therefore, it is to be understood that the invention is not to be limited to the specific embodiments disclosed and that modifications and other embodiments are intended to be included within the scope of the appended claims. Although specific terms are employed herein, they are used in a generic and descriptive sense only and not for purposes of limitation.

THAT WHICH IS CLAIMED:

1. A method for improving the flavor and aroma characters of a smoking article, comprising:
 - providing a mixture consisting essentially of a reducing sugar and optionally a hydroxide of an alkali metal;
 - subjecting said mixture to heat treatment for a time and under conditions sufficient to produce a flavorful caramel composition;
 - applying said flavorful composition to a tobacco leaf or cut filler made thereof;
 - and
 - preparing a smoking article comprising said tobacco leaf or cut filler.
2. The method of Claim 1, wherein said mixture consists essentially of a reducing sugar and a hydroxide of an alkali metal.
3. The method of Claim 2, wherein said hydroxide is sodium hydroxide.
4. The method of Claim 1, wherein said reducing sugar is fructose.
5. The method of Claim 1, wherein the heat treatment is conducted at a temperature of at least about 150°C.
6. The method of Claim 1, wherein the heat treatment is conducted at a temperature of at least about 175°C.
7. The method of Claim 1, wherein the heat treatment is conducted under a pressure of from about 20 psig to about 500 psig.
8. The method of Claim 1, wherein the heat treatment is conducted for a period of at least about 10 minutes.

9. The method of Claim 1, wherein said flavorful composition is applied at an amount of from about 5% to about 8% by weight based on the total dry weight of the tobacco leaf or cut filler in the smoking article.

10. A method for improving the flavor and aroma characters of a smoking article, said method comprising:

providing a mixture consisting essentially of high fructose corn syrup and sodium hydroxide;

subjecting said mixture to heat treatment for a time and under conditions sufficient to produce a flavorful composition;

applying said flavorful composition directly to a tobacco material selected from the group consisting of tobacco leaves and cut fillers made thereof; and

preparing a smoking article comprising said tobacco material.

11. The method of Claim 10, wherein the heat treatment is conducted at a temperature of at least about 175°C.

12. The method of Claim 10, wherein said flavorful composition is applied at an amount of from about 5% to about 8% by weight based on the total dry weight of the tobacco material in the smoking article.

13. A smoking article prepared by the process of Claim 1.

14. A smoking article prepared by the process of Claim 10.

METHOD OF IMPROVING FLAVOR IN SMOKING ARTICLE

ABSTRACT OF THE DISCLOSURE

A method for improving the flavor and aroma characters of a smoking article is provided. A mixture consisting essentially of a reducing sugar and optionally a hydroxide of an alkali metal is subjected to heat treatment for a time and under conditions sufficient to produce a flavorful composition. The flavorful composition is then applied to tobacco leaves or cut filler made thereof, and smoking articles are prepared from the tobacco leaves or cut filler.

CLT01/4394549v1

Please type a plus sign (+) inside this box → +

PTO/SB/01 (12-97)

Approved for use through 9/30/00. OMB 0651-0032

Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number

**DECLARATION FOR UTILITY OR
DESIGN
PATENT APPLICATION
(37 CFR 1.63)**

☒ Declaration Submitted with Initial Filing OR ☐ Declaration Submitted after Initial Filing (surcharge (37 CFR 1.16 (e)) required)

Attorney Docket Number	JJ-124B-R&D
First Named Inventor	Michael Francis Dube
COMPLETE IF KNOWN	
Application Number	/
Filing Date	July 25, 2000
Group Art Unit	
Examiner Name	

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

Method of Improving Flavor in Smoking Article

the specification of which (Title of the Invention)

☒ is attached hereto
OR

☐ was filed on (MM/DD/YYYY) as United States Application Number or PCT International

Application Number **TO BE ASSIGNED** and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56.

I hereby claim foreign priority benefits under 35 U.S.C. 119(a)-(d) or 365(b) of any foreign application(s) for patent or inventor's certificate, or 365(a) of any PCT international application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT international application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached?	
				YES	NO
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>
			<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto:

I hereby claim the benefit under 35 U.S.C. 119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	<input type="checkbox"/> Additional provisional application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

[Page 1 of 2]

Burden Hour Statement: This form is estimated to take 0.4 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

Please type a plus sign (+) inside this box → ☐

PTO/SB/01 (12-97)
Approved for use through 9/30/00. OMB 0651-0032

Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

DECLARATION — Utility or Design Patent Application

I hereby claim the benefit under 35 U.S.C. 120 of any United States application(s), or 365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of 35 U.S.C. 112, I acknowledge the duty to disclose information which is material to patentability as defined in 37 CFR 1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application or PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority data sheet PTO/SB/02B attached hereto.

As a named inventor, I hereby appoint the following registered practitioner(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Customer Number → ☐ Registered practitioner(s) name/registration number listed below

Place Customer
Number Bar Code
Label here

Name	Registration Number	Name	Registration Number
Grover M. Myers	25,359		
August J. Borschke	30,539		
Stephen M. Bodenheimer, Jr.	28,932		

☐ Additional registered practitioner(s) named on supplemental Registered Practitioner Information sheet PTO/SB/02C attached hereto.

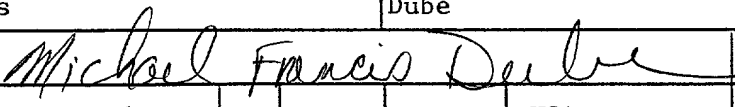
Direct all correspondence to: ☐ Customer Number OR ☒ Correspondence address below

Name	Grover M. Myers, Esq.				
Address	Law Department - Patents				
Address	R.J. Reynolds Tobacco Company, P.O. Box 1487, 950 Reynolds Blvd.				
City	Winston-Salem	State	NC	ZIP	27102-1487
Country	USA	Telephone	(336) 741-2694	Fax	(336) 741-5449

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor:

☐ A petition has been filed for this unsigned inventor

Given Name (first and middle (if any))		Family Name or Surname			
Michael Francis		Dube			
Inventor's Signature				Date	7/20/00
Residence: City	Winston-Salem	State	NC	Country	USA
				Citizenship	USA
Post Office Address	130 Northpond Lane				
Post Office Address	Winston-Salem				
City	Winston-Salem	State	NC	ZIP	27106
				Country	USA

☒ Additional inventors are being named on the 2 supplemental Additional Inventor(s) sheet(s) PTO/SB/02A attached hereto

Please type a plus sign (+) inside this box → +

PTO/SB/02A (3-97)

Approved for use through 9/30/98. OMB 0651-0032
Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

DECLARATION	ADDITIONAL INVENTOR(S) Supplemental Sheet Page <u>1</u> of <u>2</u>
--------------------	---

Name of Additional Joint Inventor, if any:		<input type="checkbox"/> A petition has been filed for this unsigned inventor					
Given Name (first and middle [if any])				Family Name or Surname			
Luis Mayan				Dominguez			
Inventor's Signature	<i>Luis Mayan Dominguez</i>				Date	7/20/00	
Residence: City	Winston-Salem	State	NC	Country	USA	Citizenship	USA
Post Office Address	230 Lucerne Lane						
Post Office Address	Winston-Salem						
City	Winston-Salem	State	NC	ZIP	27104	Country	USA
Name of Additional Joint Inventor, if any:		<input type="checkbox"/> A petition has been filed for this unsigned inventor					
Given Name (first and middle [if any])				Family Name or Surname			
Chi-Kuen				Shu			
Inventor's Signature	<i>Chi-Kuen Shu</i>				Date	7/20/00	
Residence: City	Pfafftown	State	NC	Country	USA	Citizenship	USA
Post Office Address	4409 Lochurst Drive						
Post Office Address	Pfafftown						
City	Pfafftown	State	NC	ZIP	27040	Country	USA
Name of Additional Joint Inventor, if any:		<input type="checkbox"/> A petition has been filed for this unsigned inventor					
Given Name (first and middle [if any])				Family Name or Surname			
Elwood Lee				Williams Jr.			
Inventor's Signature	<i>Elwood Lee Williams Jr.</i>				Date	7-20-00	
Residence: City	Lewisville	State	NC	Country	USA	Citizenship	USA
Post Office Address	206 Flintridge Drive						
Post Office Address	Lewisville						
City	Lewisville	State	NC	ZIP	27023	Country	USA

Burden Hour Statement: This form is estimated to take 0.4 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.

Please type a plus sign (+) inside this box → +

PTO/SB/02A (3-97)

Approved for use through 9/30/98. OMB 0651-0032
Patent and Trademark Office; U.S. DEPARTMENT OF COMMERCE

Under the Paperwork Reduction Act of 1995, no persons are required to respond to a collection of information unless it contains a valid OMB control number.

DECLARATION

ADDITIONAL INVENTOR(S)
Supplemental Sheet
Page 2 of 2

Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name (first and middle [if any])				Family Name or Surname			
Kenneth Wayne				Smith			
Inventor's Signature	<i>Kenneth Wayne Smith</i>			Date	07/20/00		
Residence: City	Winston-Salem	State	NC	Country	USA	Citizenship	USA
Post Office Address	253 Creekbend Drive						
Post Office Address	Winston-Salem						
City	Winston-Salem	State	NC	ZIP	27103	Country	USA
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name (first and middle [if any])				Family Name or Surname			
William Monroe				Coleman III			
Inventor's Signature	<i>William Monroe Coleman III</i>			Date	7/20/00		
Residence: City	Winston-Salem	State	NC	Country	USA	Citizenship	USA
Post Office Address	2900 Loch Drive						
Post Office Address	Winston-Salem						
City	Winston-Salem	State	NC	ZIP	27106	Country	USA
Name of Additional Joint Inventor, if any:				<input type="checkbox"/> A petition has been filed for this unsigned inventor			
Given Name (first and middle [if any])				Family Name or Surname			
Inventor's Signature				Date			
Residence: City		State		Country		Citizenship	
Post Office Address							
Post Office Address							
City		State		ZIP		Country	

Burden Hour Statement: This form is estimated to take 0.4 hours to complete. Time will vary depending upon the needs of the individual case. Any comments on the amount of time you are required to complete this form should be sent to the Chief Information Officer, Patent and Trademark Office, Washington, DC 20231. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Assistant Commissioner for Patents, Washington, DC 20231.